

*TRIAL OF A METHOD FOR IMPROVING
THE DURABILITY OF CHERT*

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*Joint
Highway
Research
Project*

by

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LAFAYETTE INDIANA*

INFORMATIONAL REPORT

TRIAL OF A METHOD FOR IMPROVING THE DURABILITY OF CEMENT

TO: E. B. Woods, Director
Joint Highway Research Project

FROM: H. L. Michael, Assistant Director
Joint Highway Research Project

October 23, 1958

File: 5-4-2

Attached is an informational report entitled, "Trial of a Method for Improving the Durability of Cement," by Mr. Sander Ponovits, Graduate Assistant on our staff. The report and research has been conducted under the supervision of Dr. J. F. McLaughlin, Research Engineer on our staff.

This research is another effort of the Project to provide information on aggregates for the purpose of improving portland cement concrete. This report deals with improving the durability of cement. It is presented to the Board for information.

Respectfully submitted,

H. L. Michael

H. L. Michael, Assistant Director
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HLM:acc

Attachment

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INFORMATIONAL REPORT

TRIAL OF A METHOD
FOR
IMPROVING THE DURABILITY OF CEMENT

By

Sender Popovics
Graduate Assistant

Joint Highway Research Project
Project No. G-36-18B
File No. 5-4-2

Purdue University
Lafayette, Indiana

October 23, 1958

TRIAL OF A METHOD FOR IMPROVING THE DURABILITY OF CHERT

by

G. Popovics, Graduate Assistant

PURPOSE OF THE TEST

It is well known that there are some kinds of chert which are not suitable for making concrete because they may cause deterioration of the concrete when it is subjected to freezing and thawing. According to existing research, the cause of this is that these chert particles do not have adequate durability because of the nature of their pore systems. Consequently, two methods offer themselves for stopping this injurious effect:

a. The chert particles of poor quality should be removed from the aggregate.

To accomplish this, the fact that the specific gravity of the chert particles of poor quality is lower than 2.55 can be utilized. Thus, these particles can be separated by a centrifugal machine or by means of some liquid of high specific gravity. This method has been already employed on a number of jobs.

b. A simple treatment should be done to chert particles. For this treatment, it is required that, as a result, the water absorption of chert decreases by a still harmless value; but simultaneously, this treatment should not have any harmful effect on the concrete made with the treated chert. This method might be needed in addition to the method mentioned above in (a).

because in many cases the quantity of the chert particles of poor quality is so large that the separation method is not economical.

The purpose of this test program was to try out such a simple method of treatment.

THE FUNDAMENTAL PRINCIPLES OF THE METHOD

The test method utilizes the fact that diluted solutions of sodium silicate (water-glass) and calcium chloride react with each other, the result being a solid compound; namely, calcium silicate, which is insoluble in water. The equation of the process is:



If this reaction takes place in pores of the chert, it is expected that the calcium silicate which is produced will close most of the pores; consequently, the water absorption of chert particles will decrease considerably.

Calcium silicate does not affect concrete because it is insoluble in water; sodium chloride (common salt) does not affect concrete either when in a small quantity.

MATERIALS USED

The aggregate, from which the chert was separated, was 82-16. By means of a suitable mixture of carbon tetrachloride ($\gamma = 1.58$) and acetylenetetrabromide ($\gamma = 2.97$), those particles, which had a specific gravity of less than 2.50, were separated from the aggregate and from those particles the chert particles were selected by visually. The tests were then carried out with these chert particles.

For testing the chert particles, technical grade water-glass of $40^{\circ} - 42^{\circ} \text{Bé}$ and anhydrous calcium chloride were used. The concentration of the solutions used was generally 10 percent. This meant the following:

- a. To 1 unit weight of water-glass, 3 unit weights of water were added
- b. To 1 unit weight of calcium chloride, 9 unit weights of water was added

The temperature of the solutions was generally $22-23^{\circ}\text{C}$.

METHOD OF TEST AND TESTING RESULTS

The samples of chert were dried out after which they were placed into solutions of calcium chloride and water-glass in various ways, and finally the water absorption was determined again. The difference between the values of the water absorption before and after the treatment was considered as a measure of the effectiveness of the treatment. The plan was that a bigger quantity of chert would be treated with the most efficient methods and durability tests of concrete made with these treated cherts will be carried out.

The results of the performed tests are reported with a few exceptions, in chronological order. The basic idea of the test in question is explained briefly before presenting the results of the test.

Treatment First with Solution of Water-glass

The first experiment was carried out in the simplest way, as follows: the chert sample was placed into the solution of water-glass for 10 seconds, immediately after this it was placed into the solution of calcium chloride for 10 seconds, afterwards it was held on room air for two minutes, then placed into water in order to perform the control

water absorption. The results of the tests, shown in Table I, indicate that this method of treatment is ineffective.

TABLE I

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 10 sec. in water-glass sol., 10 sec. in calc. chlor. sol., and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
1	3.80		4.00	
17	2.10	<u>2.87</u>	2.14	<u>3.05</u>
32	2.70		3.00	

Consequently, this method of treatment is very ineffective.

In the previous test chert was placed in water 2 minutes after the treatment. Theoretically this procedure should not influence the water absorption of treated chert because calcium silicate is insoluble in water. However, it was necessary to prove in the beginning of the tests whether the above assumption also is true practically. Therefore, the previous tests were repeated by keeping the samples in room air for 24 hours after the treatment and before the control water absorption, and only after this were they placed in water. The results of the test are shown in Table 2.

TABLE 2

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 10 sec. in water-glass sol., 10 sec. in calc. chlor. sol., and 24 hours in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
27	2.80		2.54	
29	4.20	<u>3.07</u>	3.43	<u>2.91</u>
6	2.20		2.73	

As was expected, the longer storage in air after treatment does not have any significance on the effect of the treatment.

Other reasons for the lack of effectiveness of the treatment might be that too small a quantity of the solution penetrates into pores in the time permitted. Therefore, in another test the samples were treated in the solution of water-glass for 3 hours. The results of this test are shown in Table 3.

TABLE 3

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 3 hrs. in water-glass sol., 10 min. in calc. chlor. sol., and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
16	10.80		10.10	
25	2.10	<u>5.44</u>	2.08	<u>5.24</u>
10	3.40		5.53	

The previous test was repeated so that the samples were held in room air for 24 hours after the treatment and before the control water absorption. The results of the test are shown in Table 4.

TABLE 4

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 3 hrs. in water-glass sol., 10 min. in calc. chlor. sol., and 24 hrs. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
13	1.80		1.74	
20	3.10	<u>2.14</u>	2.66	<u>2.23</u>
21	2.40		2.28	

The longer storage in air after treatment does not have any significance on the effect of treatment even when using the longer immersion time.

Rate of Reaction

A possible reason for failure of the tests described could be that the speed of reaction between the solutions of water-glass and calcium chloride is too high. As a result, a calcium silicate stopper is formed in the opening of pores immediately, and this hinders the solution of calcium chloride from penetrating further into the pores.

A reduction of the reaction speed can be produced in various ways. One possibility is that, after immersion in the first solution, i.e., in the water-glass solution, the particles are dried causing part of the water-glass to become solid. Therefore, when the particles are placed into the second solution, there is a reaction between the partly solid water-glass and liquid calcium chloride; the reaction speed in this case is considerably lower than that between the two solutions. Thus, the solution of calcium chloride can penetrate deeper into the pores.

Three different methods of drying were employed. The results of these tests are shown in Tables 5 - 7.

TABLE 5

Water Absorption of Plain and Treated Chert, % by Weight

Treatments: 2 hrs. in water-glass sol., the surface of
particles dried with compressed air, 1/2 hr.
in calc. chlor. sol., and 2 min. in air.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>AVG.</u>	<u>Single</u>	<u>AVG.</u>
40	7.35		5.37	
34	1.93	<u>4.31</u>	3.00	<u>3.97</u>
33	3.66		3.52	

TABLE 6

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in water-glass, 24 hrs. in room air,
1/2 hr. in calc. chlor. sol., and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
39	5.30		5.45	
46	3.54	4.04	3.52	4.01
48	3.18		3.06	

TABLE 7

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in water-glass sol., 3 hrs. at 105° C.,
1/2 hr. in calc. chlor. sol., and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
42	5.26		5.00	
44	4.35	4.81	4.83	4.91

According to these results, the drying between the two immersion does not have any influence on the effect of treatment.

Repeated Treatment

An attempt was made to increase the absorption of the solution by treating the samples with the solutions not once but several times. The results of these tests are shown in Tables 8 - 11.

TABLE 8

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in water-glass sol., 2 min. in calc.
chlor. sol., 2x (2 min. in water-glass sol.,
and 2 min. in calc. chlor. sol.), and 2 min. in air

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
18	1.50		1.09	
13	1.80	2.04	1.24	1.30
27	2.80		1.58	

TABLE 9

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in water-glass sol., 10 min. in calc. chlor. sol.,
2x (2 min. in water-glass sol., and 2 min. in calc. chlor. sol.),
and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
17	2.10		0.72	
25	2.10	2.70	1.02	1.34
9	3.90		2.29	

TABLE 10

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in water-glass sol., 1/2 hr. in calc. chlor. sol.,
2x (2 min. in water-glass sol., and 2 min. in calc. chlor. sol.),
and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
16	10.80		10.00	
21	2.40	5.53	1.74	3.00
10	3.40		3.25	

TABLE 11

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 3x (5 min. in water-glass sol. and 5 min. in calc. chlor. sol.),
and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
3	4.40		3.50	
1	3.80	3.77	3.69	3.14
20	3.10		2.22	

Tables 8 - 11 show that repetition of treatment effects a slight reduction in the water absorption of chert.

Tests of the Process of Solution Absorption

The failure of the previous trials necessitated a test to determine how much fluid penetrates into the pores of chert. Therefore, the process of water absorption and absorption of the two solutions was tested with six plain chert samples. The results of the tests are shown in Tables 12 - 14 and in Figure 1.

TABLE 12

Process of the water absorption of plain chert

Sample Designation	Water absorption, % by weight				
	1 hr.	2 hrs.	3 hrs.	24 hrs.	Vacuum
52	0.30	0.30	0.36	0.68	1.96
53	1.32	1.63	1.88	2.09	3.76
54	1.39	1.77	1.85	2.40	5.10
55	1.07	1.35	1.39	1.87	3.85
56	0.97	1.15	1.30	1.71	2.77
59	0.24	0.28	0.39	1.02	1.50

TABLE 13

Process of the water-glass sol. absorption
of plain chert

Sample Designation	Water-glass absorption, % by weight			
	1 hr.	2 hrs.	3 hrs.	24 hrs.
54	0.84	1.14	1.26	2.32
55	0.40	0.48	0.60	1.15
59	0.10	0.10	0.15	0.39

TABLE 14

Process of the calc. chlor. sol. absorption
of plain chert

Sample Designation	Calc. chlor. absorption, % by weight			
	1 hr.	2 hrs.	3 hrs.	24 hrs.
53	1.75	2.17	2.30	2.63
56	1.43	1.62	1.62	1.94
52	0.51	0.55	0.55	0.90

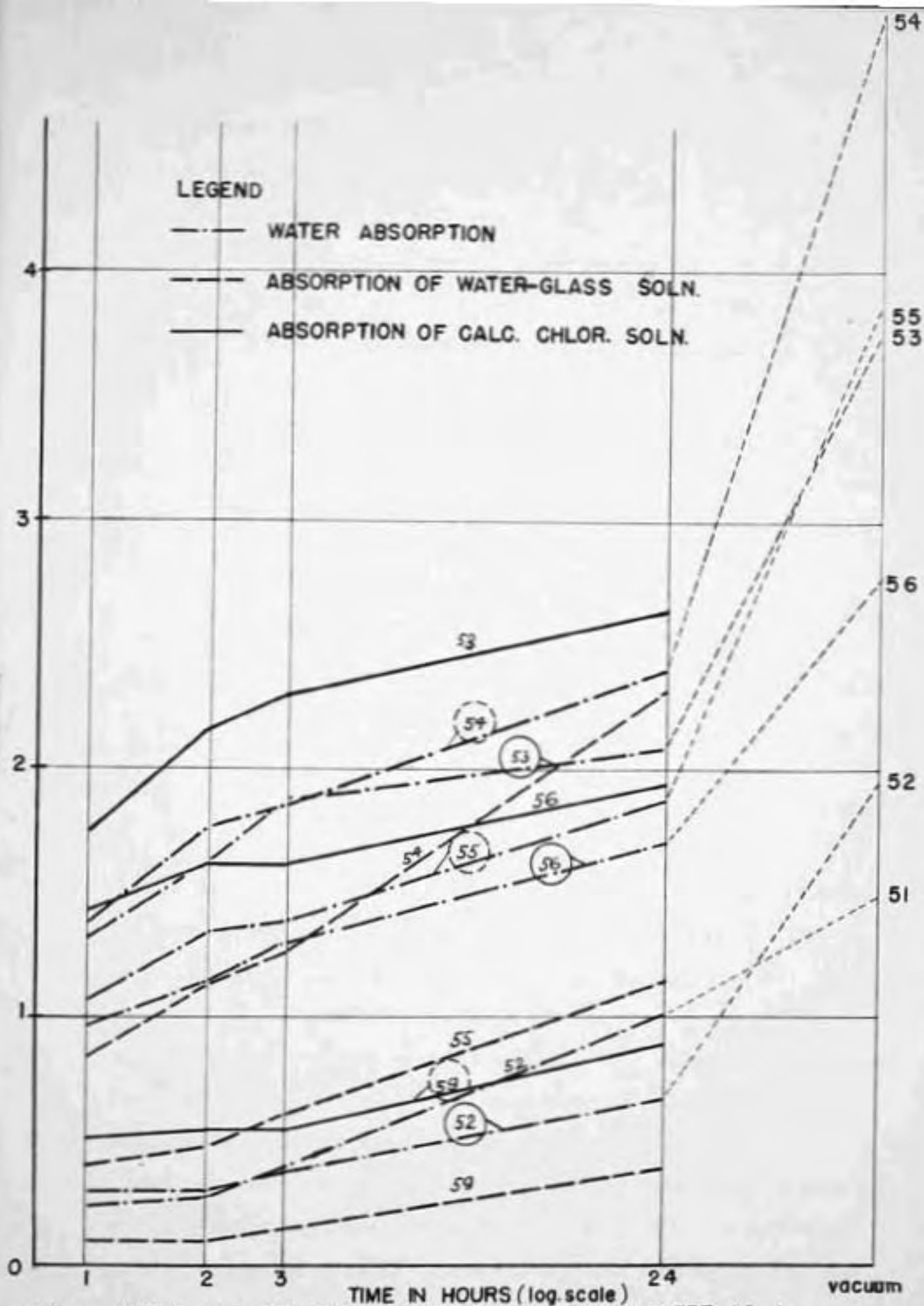


FIG. 1. WATER AND SOLUTION ABSORPTION OF PLAIN CHERT AS A FUNCTION OF TIME

The results in Tables 12 -- 14 show the following (see Figure 1):

- a. During first two hours most of the 24 hrs. fluid absorption takes place.
- b. The speed of penetration of the calcium chloride solution is higher than that of water, whereas that of the water-glass solution is lower. Consequently, the speed of penetration of the water-glass solution is much lower than that of the calcium chloride solution. In order to verify this statement, an additional test was carried out. The results of this test are shown in Table 15.

TABLE 15

Test of fluid absorption of plain chert

Sample Designation	Water absorption 1/2 by wt. 24 hrs.	Absorption of water- glass sol., 1/2 by wt.		Absorption of calc. chlor. sol., 1/2 by wt.	
		3 hrs.	24 hrs.	3 hrs.	24 hrs.
22	3.30	0.84	1.59	—	—
29	4.20	0.28	0.62	—	—
8	2.60	0.26	0.40	—	—
24	3.80	—	—	3.49	4.57
32	2.70	—	—	1.22	3.37
6	1.80	—	—	1.58	2.84

The results of Table 15 support the preceding statements concluded on the basis of Figure 1. However, it appeared from these results that the hitherto employed order of treatment 1. water-glass sol., 2. calc. chlor. sol.—was not the proper one because, in the case of equal immersion times, a larger quantity of calcium chloride solution can penetrate into the pores than can a water-glass solution. Consequently, a reversed order of immersion might result in a treatment which is more efficient.

The order of plunge employed hitherto, however, was not accidental. Some free material always remains from the last solution on surface of particles. Calcium chloride is quite neutral for concrete when in such

a small quantity, but a small quantity of water-glass can easily cause a disagreeable localized flash set. In order to avoid this, the immersion order mentioned first was chosen.

Treatment First with Solution of Calcium Chloride

On the basis of the results shown in Tables 12 - 15, the chert particles were further treated, first with a calcium chloride solution for various times and then with a water-glass solution. After this, it was necessary to neutralize the free water-glass, by a quick immersion in calcium chloride. The results of the tests are shown in Tables 16 - 21.

TABLE 16

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 10 sec. in calc. chlor. sol., 10 sec. in
water-glass sol., 10 sec. in calc. chlor. sol.,
and 2 min. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
22	3.30		2.27	
3	4.40	3.43	3.13	2.55
8	2.60		2.26	

TABLE 17

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 10 sec. in calc. chlor. sol., 10 sec. in
water-glass sol., 10 sec. in calc. chlor. sol.,
and 24 hrs. in air.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
18	1.50		1.55	
24	3.80	3.07	3.53	3.13
9	3.90		4.31	

TABLE 18

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 10 min. in calc. chlor. sol., 10 min. in

water-glass sol., 2 min. in calc. chlor. sol.,

and 2 min. in air.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>Avg.</u>	<u>Single</u>	<u>Avg.</u>
40	7.35		5.65	
42	5.26	<u>5.57</u>	5.12	<u>5.39</u>
51	4.09		5.20	

TABLE 19

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 30 min. in calc. chlor. sol., 10 min. in

water-glass sol., 2 min. in calc. chlor. sol.,

and 2 min. in air.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>Avg.</u>	<u>Single</u>	<u>Avg.</u>
39	5.30		5.48	
61	1.89	<u>4.05</u>	4.12	<u>4.04</u>
64	2.96		2.52	

TABLE 20

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 1 hr. in calc. chlor. sol., 10 min. in

water-glass sol., 2 min. in calc. chlor. sol.,

and 2 min. in air.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>Avg.</u>	<u>Single</u>	<u>Avg.</u>
48	3.18		2.92	
57	3.71	<u>3.33</u>	3.99	<u>3.36</u>
60	3.10		3.16	

TABLE 21

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 2 hrs. in calc. chlor. sol., 10 min. in

water-glass sol., 2 min. in calc. chlor. sol.,

and 2 min. in air.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>Avg.</u>	<u>Single</u>	<u>Avg.</u>
33	3.66		3.69	
44	4.35	3.72	4.13	3.75
58	3.15		3.42	

It is seen from the results shown in Tables 16 - 21 that even the reversed order of immersion does not decrease the water absorption significantly.

Treatment by Means of Vacuum

In order to increase the solution absorption, chert particles were saturated with the solution of calcium chloride by means of vacuum; after which they were placed in the solution of water-glass. The results of the test are shown in Table 22.

TABLE 22

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),

16 hrs. in water-glass sol., 2 min. in

calc. chlor. sol., and 16 hrs. at 105° C.

<u>Sample Designation</u>	<u>Plain</u>		<u>Treated</u>	
	<u>Single</u>	<u>Avg.</u>	<u>Single</u>	<u>Avg.</u>
57	3.71		3.06	
71	1.87	3.06	1.76	2.83
78	3.60		3.82	

Some decrease in water absorption appears but more is desirable.

In order to slow the chemical reaction, some chert samples were dried in various ways after the treatment with calcium chloride solution in vacuum and before the treatment with water-glass. The results of the test are shown in Tables 23 - 25.

TABLE 23

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),

24 hrs. in air, 16 hrs. in water-glass sol.,

2 min. in calc. chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
72	2.81		1.94	
44	4.35		2.00	
58	3.15		1.82	
53	2.09	<u>2.42</u>	1.35	<u>1.22</u>
54	2.40		0.83	
55	1.87		0.78	
56	1.71		0.91	
59	1.02		0.10	

TABLE 24

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),

the surface of particles dried with compressed air,

16 hrs. in water-glass sol., 2 min. in calc.

chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
60	3.10		1.77	
65	3.23	<u>2.77</u>	2.35	<u>2.17</u>
66	1.97		1.89	

TABLE 25

Water Absorption of Plain and Treated Chert, % by Weight
 Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),
 2 hrs. at 105°C., after cooling down 16 hrs. in
 water-glass sol., 2 min. in calc. chlor. sol.,
 and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
6	1.80		1.03	
64	2.96	3.52	2.32	2.32
86	5.80		3.60	

TABLE 26

Water Absorption of Plain and Treated Chert, % by weight
 Treatment: 24 hrs. in calc. chlor. sol. (with vacuum).
 24 hrs. at 105°C., after cooling down 16 hrs. in
 water-glass sol., 2 min. in calc. chlor. sol.,
 and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
74	3.02		1.38	
32	2.70	2.86	0.20	0.79

As a result of the vacuum saturation, some decrease appears in water absorption, however, a greater decrease is desirable.

Another possibility of decreasing the reaction speed is to reduce the concentration of one of the solutions. It would be desirable to keep the concentration of the calcium chloride solution at 10 percent, and reduce the concentration of the water-glass solution to 5 percent. The results of these tests are shown in Tables 27 and 28.

TABLE 27

Water Absorption of Plain and Treated Chert, % by Weight
 Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),
 the surface of particles dried with compressed air,
 16 hrs. in water-glass sol. of concentration of 5%,
 2 min. in calc. chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
22	3.30		1.20	
61	3.69	3.68	2.90	2.24
69	3.85		2.58	

TABLE 28

Water Absorption of Plain and Treated Chert, % by Weight
 Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),
 24 hrs. at 105°C., after cooling down 16 hrs. in
 water-glass sol. of concentration of 5%, 2 min.
 in calc. chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
82	6.28		5.78	
84	4.51	4.30	3.84	3.23
25	2.10		0.33	

As a result of this treatment, some decrease appears for water absorption in Tables 27 and 28, but a greater decrease is desirable.

It is possible to assist the penetration of the water-glass solution into the pores in such a way that the chert, treated with calcium chloride solution and then dried, is placed into the water-glass solution in a warm state. Because of the contraction of cooling air in the pores, the water-glass solution can penetrate into the chert in a larger quantity than if the particles had received the solution in cool state. The results of the tests are shown in Tables 29 and 30.

TABLE 29

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),

24 hrs. at 105°C., in warm state 16 hrs. in water-

glass sol. of concentration of 10%, 2 min. in

calc. chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
9	3.90		0.20	
29	4.20	3.76	0.00	0.38
48	3.18		0.94	

TABLE 30

Water Absorption of Plain and Treated Chert, % by Weight

Treatment: 24 hrs. in calc. chlor. sol. (with vacuum),

24 hrs. at 105°C., in warm state 16 hrs. in water-

glass sol. of concentration of 5%, 2 min. in

calc. chlor. sol., and 16 hrs. at 105°C.

Sample Designation	Plain		Treated	
	Single	Avg.	Single	Avg.
21	2.40		1.00	
40	7.35	5.00	4.23	2.19
42	5.26		1.34	

As a result of this treatment, a considerable decrease in water absorption was obtained in Tables 29 and 30.

SUMMARY

It can be seen from the test results that treating chart with solutions of calcium chloride and water-glass reduces the water absorption considerably only if the saturation is assisted by vacuum and heat. The degree of reduction of water absorption is not enough by other methods used in this study.